

TECHNICAL NOTE: COMPARISON OF ACCELERATED METHODS FOR EVALUATING LEACHING FROM PRESERVATIVE-TREATED WOOD¹

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Abstract. Current standardized methods are not well-suited for estimating in-service preservative leaching from treated wood products. This study compared several alternative leaching methods to a commonly used standard method, and to leaching under natural exposure conditions. Small blocks or lumber specimens were pressure treated with a wood preservative containing borax and copper hydroxide. The specimens were leached using scenarios involving short periods of immersion, simulated rainfall, or to a longer period of outdoor exposure to natural precipitation. When compared with lumber specimens exposed to natural precipitation, leaching from immersed small blocks overestimated losses of both copper and of boron, whereas immersion of lumber specimens underestimated losses of copper. Stirring during immersion, which is required by some standard methods, did not affect leaching. Simulated rainfall most closely simulated leaching during outdoor exposure, but is relatively complex and may be difficult to standardize. Leaching appeared to be directly related to the time that specimens had sufficient moisture to allow diffusion to occur. Further research is needed to better characterize moisture contents of wood products outdoors and develop methods that simulate those moisture conditions.

Keywords: Wood preservative, leaching, methods, boron, copper, rainfall, moisture content.

INTRODUCTION

The resistance of wood preservatives to leaching from treated wood is important in evaluating both the efficacy and potential environmental impact of wood preservatives. However, obtaining useful and representative estimates of preservative leaching has proved challenging. This is especially the case for treated wood used aboveground or abovewater, where leaching is a function of precipitation. Although the most realistic leaching estimates are obtained by expos-

ing treated wood to in-service conditions, this approach requires lengthy exposures and is often not reproducible.

Conventional laboratory leaching methods were primarily developed to allow comparison between experimental formulations and use small specimens to accelerate leaching (Lebow et al 2008). In the United States, the most commonly used standardized leaching method for preservative-treated wood is AWP Method E11-12, Standard Method for Accelerated Evaluation of Preservative Leaching (AWPA 2015). This method specifies treatment of small (19 mm) blocks, which are subsequently vacuum impregnated with water, then remain immersed with agitation, for 14-17 da. The higher proportion of exposed end

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grain in the small blocks is expected to accelerate leaching. Although this method is not intended for use in estimating release from in-service-treated products, it has sometimes been used for this purpose in the absence of other methods. The Japanese (JIS K 1571) and Chinese (CNS 6717) leaching methods use weathering steps as specimen preparation for exposure to biological attack (CNS 2000; JSA 2004). Again, the small size and grain orientation ($10 \times 20 \times 20$ mm with the 10 mm parallel to the grain) of the specimens is expected to greatly accelerate leaching. A European method (EN 84) is also intended as a conditioning step before biological exposure (BSI 1997). It uses somewhat larger specimens ($15 \times 25 \times 50$ mm) with a lower proportion of end grain than the United States, Japanese, or Chinese methods. Unlike the United States, Japanese, and Chinese methods, EN 84 does not specify agitation during leaching. The Organization for Economic Co-operation and Development (OECD) has also developed guidelines for evaluating biocide release from preservative-treated wood, and these methods are intended for use in estimating release from in-service products. Separate methods are recommended for wood intended for immersion vs applications aboveground or abovewater. For wood used aboveground, OECD guidelines describe an approach involving a brief dip immersion using small ($15 \times 25 \times 50$ mm) specimens (OECD 2009). The dip immersions can be either three 1-min dips, two 1-h dips, or one 2-h dip per day for 19 da. Although intended to simulate in-service leaching, there is some concern that this approach may not represent commercially produced lumber (Baines 2005) or not produce the moisture conditions reported for wood products exposed to natural weathering (Lebow et al 2008). One study which compared outdoor leaching to the OECD method concluded that the laboratory method risked underestimating in-service leaching (Morsing and Lindegaard 2004).

Another approach to evaluating leaching from wood exposed to precipitation is simulated rainfall (Cooper and MacVicar 1995; Lebow et al 2003; Lebow et al 2004; Morrell et al 2004;

Mitsuhashi et al 2007). Simulated rainfall can create more realistic wetting conditions, and allows some control over rainfall rates and frequency. However, the equipment required is more complex than that required for other laboratory leaching methods, and none of these approaches have been standardized. In addition, although volumes and frequency of rainfall events can be accelerated with simulated rainfall, the effect of diffusion periods between rainfall events is more difficult to accelerate.

A limitation of current laboratory leaching methods is their lack of consideration of wood moisture content (MC) relative to that of treated wood exposed aboveground. With the exception of methods that specify initial saturation of the wood via vacuum impregnation (eg AWP A E11) the methods do not attempt to control moisture conditions, or even track the resulting wood MC. This may in part result from a lack of understanding of actual in-service MCs. Many factors can affect the MC of exposed wood products, including local climate conditions, dimensions of member, degree of checking, orientation (eg vertical or horizontal) and type of preservative treatment. Researchers have reported that the MC of pine sapwood exposed to natural weathering may range from maximums of 80% to minimums of approximately 10% (Belford and Nicholson 1969; Edlund and Sundman 1989; Militz et al 1998; Rapp et al 2000; Lindegaard and Morsing 2003; Hedley et al 2004; Saladis and Rapp 2004; Rydell et al 2005). Most of the maximums reported fell within 40-55% for horizontal specimens and between 30% and 50% for vertical specimens. Minimum MSs generally fell into the 10-15% range. Average MSs reported for horizontal exposures ranged from 21% to 26%, whereas the averages reported for vertical exposure were 18.6% and 25.4%. MSs reported for less permeable species, such as spruce or Douglas-fir, tended to be lower than those of pine species when exposed under similar conditions (Lindegaard and Morsing 2003; Hedley et al 2004; Brischke and Rapp 2008).

Current standardized leaching methods are not well suited for estimating in-service leaching,

particularly for aboveground structures. In part, this may be because these methods do not attempt to simulate the MSs that may occur in wood exposed to precipitation. In this study, we compared several alternative leaching methods to a commonly used standard method, and to leaching under natural exposure conditions. Specimen MC was evaluated and compared with that observed for specimens exposed outdoors.

MATERIALS AND METHODS

Specimen Preparation

Specimens were prepared from five southern pine parent boards with dimensions of $38 \times 92 \times 2438$ mm ($2 \times 4 \times 8$ ft nominal). Specimens were selected to be free of heartwood, knots, and other visible wood defects. Matching sets 19-mm cubes and $38 \times 92 \times 102$ mm long lumber sections were cut from each parent board.

All specimens were conditioned to constant weight at 23°C and 55% RH before preservative treatment. The lumber specimens were end sealed with two coats of neoprene rubber sealant to prevent end-grain penetration during preservative treatment as well as subsequent leaching from the end grain.

Preservative Treatment Process

The preservative evaluated in this study was an alkaline borax-copper formulation containing 1.3% elemental boron and 0.5% elemental copper. This formulation was selected because it contains a readily leachable component (boron)

as well as a less leachable component (copper). It is important to note that this formulation is not currently used for commercial pressure treatments, and that the quantities of preservative leached reported in this study are not directly applicable to any current commercial pressure treatment preservatives. A full cell treatment schedule was used to enhance uniformity of treatment. An initial 30-min vacuum at -81 kPa (gauge) was followed by introduction of the treatment solution and a 60-min pressure period at 1034 kPa (gauge). The specimens were weighed before and after treatment to allow calculation of preservative uptake (Table 1). Following treatment the specimens were stored in plastic bags for 1 wk to prevent rapid drying and then reequilibrated in a room maintained at 23°C and 55% RH.

Leaching Conditions Evaluated

To minimize the effects of wood properties on leaching, specimens were assigned so that each parent board was equally represented in each leaching scenario. The leaching conditions are summarized in Table 2.

1. BlockStir: This method is American Wood Protection Association Standard E11-12, Standard Method for Accelerated Evaluation of Preservative Leaching (AWPA 2015). This is currently the most commonly used standardized leaching method in North America. In this method, six treated 19-mm blocks are placed into each leaching container, weighted to prevent floating, and vacuum impregnated

Table 1. Uptake of elemental boron and copper in specimens used for each leaching condition.

Leaching condition	Uptake (g)				Uptake (kg/m ³)			
	Boron		Copper		Boron		Copper	
	Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation
BlockStir ^a	0.37	0.02	0.14	0.01	9.10	0.43	3.50	0.17
BlockNoStir ^a	0.38	0.02	0.14	0.01	9.12	0.43	3.51	0.16
LumStir	2.91	0.34	1.12	0.13	9.21	1.08	3.54	0.41
LumNoStir	2.90	0.29	1.11	0.11	9.17	0.92	3.53	0.35
LumSim	2.90	0.29	1.12	0.11	9.20	0.93	3.54	0.36
LumOut	2.98	0.24	1.15	0.09	9.43	0.78	3.63	0.30

^a Based on the combined uptake of the six blocks in each of the five replicate flasks.

Table 2. Summary of leaching conditions evaluated.

Designation	Leaching condition	Specimen dimensions	No. of reps	Duration (da)	Number and timing of leachate collections
BlockStir (standard)	Immersion with stirring	6, 19-mm blocks	5	16	9 (0.25, 1, 2, 4, 7, 9, 11, 14, and 16 da)
BlockNoStir	Immersion without stirring	6, 19-mm blocks	5	16	9 (0.25, 1, 2, 4, 7, 9, 11, 14, and 16 da)
LumStir	Immersion with stirring	38 × 92 × 102 mm	5	16	9 (0.25, 1, 2, 4, 7, 9, 11, 14, and 16 da)
LumNoStir	Immersion without stirring	38 × 92 × 102 mm	5	16	9 (0.25, 1, 2, 4, 7, 9, 11, 14, and 16 da)
LumSim	Simulated rainfall	38 × 92 × 102 mm	10	25	8 (2, 4, 9, 11, 16, 18, 23 and, 25 da)
LumOut	Outdoors, natural weathering	38 × 92 × 102 mm	5	268	8 (39, 67, 103, 116, 151, 182, 224, and 268 da, based on rainfall received)

with 300 mL of deionized water. The small blocks, with their relatively high proportion of exposed end grain, are intended to accelerate leaching. Five replicate containers, or a total of thirty 19-mm blocks were used in this study. The containers were placed on an orbital shaker and agitated at 60 rpm. The leaching water was collected and replaced after 0.25, 1, 2, 4, 7, 9, 11, 14, and 16 da.

2. BlockNoStir: This method is identical to that of Blockstir, except that the leaching containers were not placed on an orbital shaker.
3. LumStir: This method was identical to that of Blockstir, except a single, 38 × 92 × 102 mm lumber specimen was used instead of six smaller blocks. The end grain of the specimens had also been sealed with a neoprene rubber coating to limit leaching to the radial and tangential surfaces. A larger leaching container was used and the volume of leaching water was increased to 600 mL in proportion to the increased surface area.
4. LumNoStir: This method was identical to that of Lumstir, except that the leaching containers were not placed on an orbital shaker.
5. LumSim: This method used simulated rainfall to leach 38 × 92 × 102 mm lumber specimens. Ten specimens (two from each parent board) were placed into separate stainless steel trays, which were slightly wider and longer (98 × 108 mm) than the specimens. The specimens were supported on a plastic grid so that they were above the tray outlet drain. Run-off from the specimens

drained through tubing into polyethylene collection containers below. Simulated rainfall (run-off water) was applied from a rotating fan-spray nozzle mounted 1 m above the specimens. The rate of rainfall was controlled at 8 mm/h by the speed of the nozzle sweep and by cycling the nozzle off and on during rainfall events. Daily rainfall was applied in 60-min intervals (60 min on, 60 min off) over 13 h (a total of 7 h of rainfall per day). Rainfall was applied 4 da/wk (Monday to Thursday). The run-off from the specimens was collected twice per week, after 112 mm of rainfall had accumulated. This pattern was repeated for 4 wk, yielding a total of 896 mm of rainfall and eight leachate collections.

6. LumOut: Five lumber specimens (38 × 92 × 102 mm) were placed into stainless steel trays (98 × 108 mm) with drain hoses. The specimens were exposed outdoors to natural rainfall from March 6 to November 29 at a site near Madison, WI. A weather station installed adjacent to the specimens collected rainfall data at 15-min intervals. The specimens were exposed to 868 mm of rainfall, and leachate was collected eight times, after rainfall accumulations of 113, 105, 114, 121, 174, 102, 92, and 45 mm. The intent was to collect rainfall after approximately 112 mm of accumulation, corresponding to the collection intervals used in the simulated rainfall methodology. However, the final leaching period was curtailed because of sustained subfreezing temperatures.

MC Measurements

A resistance type moisture meter was used to evaluate the internal MC of the LumSim and LumOut specimens (specimens leached by other methods were initially vacuum impregnated with water and thus had MSs in excess of 100%). The moisture meter used in this study was a General Electric Protimeter Timbermaster (General Electric Sensing, Danbury, CT), which displays MC readings between 7% and 100%. The internal calibration recommended for southern pine was used in this study. Stainless steel screws were used as electrodes because preliminary trials indicated that the pin electrodes tended to yield lower, and more variable, MC readings. Initially, 10-mm-diameter holes were drilled to depth of 19 mm into the center of a narrow face of each specimen and filled with silicone caulk. After the caulk dried, trim head wood screws (no. 7, 76 mm length) were driven through the caulk and into the specimen until they extended to within 19 mm of the opposite narrow face. Pilot holes were used to ensure that the screws remained aligned as they were driven into the wood. The two screws, spaced 25 mm apart, were thus measuring the MC in an internal zone that was approximately 39 mm from each end and 19 mm from the wide and narrow faces of the specimens.

The copper hydroxide and borax retention in the specimens was relatively high, and preliminary trials indicated that an adjustment was needed to correct the resistance readings. The adjustment was developed by treating small specimens of southern pine sapwood with the same retention of preservative, and then equilibrating those specimens to a range of MSs. Matched specimens treated with deionized water were included for comparison. Immediately after meter readings were taken, the specimens were weighed and then oven-dried to allow calculation of gravimetric MC. The data were used to develop a segmented linear regression model, with censoring and heterogeneity between segments. The MC readings reported in this article have been adjusted according to this model. This procedure also indicated that adjusted MC read-

ings above 40% were poorly correlated to oven-dry MC.

Temperature may also affect resistance readings, and a temperature correction was developed for the LumOut specimens. In this case, the leaching specimens were used to develop the correction. The specimens were wrapped in plastic to prevent drying and equilibrated at set temperatures ranging from 4°C to 31°C. The readings were then adjusted to those obtained at 20°C.

Analysis of Leachate Solutions

The collected leachate was acidified to below pH 2 with nitric acid to maintain copper solubility and analyzed by inductively coupled plasma emission spectrometry.

RESULTS AND DISCUSSION

As expected, the percent of boron and copper leached was greatest from the small (19 mm) blocks (Fig 1). In addition to their small size, these blocks have a high proportion of exposed end grain. Because the rate of movement of liquids along the grain of wood is several orders of magnitude greater than that across the grain, greater leaching is likely to occur from shorter dimensions with a higher proportion of end grain. This effect was especially noticeable for the more leachable component boron, which was over 90% depleted after only four leachate collections (Fig 1).

Losses were more gradual for the larger lumber specimens with sealed end grain. For boron, the simulated rainfall exposure caused the greatest percentage leaching from lumber specimens, followed by the immersed lumber and outdoor exposure specimens. However, the four exposures produced surprisingly similar results by the eighth leachate collection, with cumulative boron losses of 74%, 71%, 70%, and 65% for the LumSim, LumStir, LumNoStir, and LumOut specimens, respectively (Fig 1). Copper loss from the lumber specimens varied more greatly by leaching method, with the LumStir and

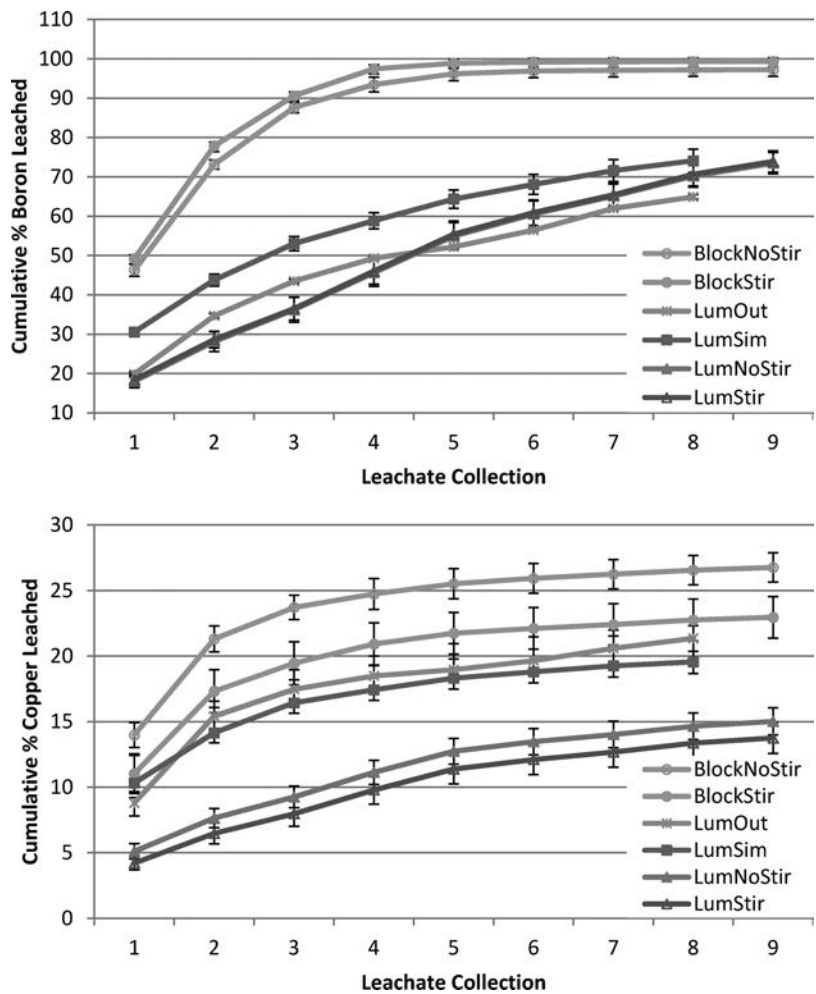


Figure 1. Average cumulative percentage of boron (upper) or copper (lower) leached from specimens. Error bars represent one standard deviation from the mean.

LumNoStir methods producing substantially less cumulative leaching than the LumSim or LumOut exposures. This finding may appear counterintuitive, but it is important to note that the time between collection intervals was greater for the LumSim specimens during the early stages of leaching, and much greater for the LumOut specimens for all collection intervals. Previous studies using specimens exposed outdoors have noted that the interval between rainfall events appears to influence leaching, with greater amounts leached after longer resting periods. This effect has been attributed to the allowance for a longer period for soluble preservative com-

ponents to diffuse to the surface from the interior of the wood products (Chung and Ruddick 2004; García-Valcarcel et al 2004; Taylor and Cooper 2005; Ruddick 2008; Hasan et al 2010; Waldron and Cooper 2010; Tao et al 2013). The amount of leaching that occurred can also be compared as a function of the time that the specimens were in contact with the leaching water. For the LumSim and LumOut specimens, this time was considered as hours of rainfall. Hours of rainfall was calculated for the LumOut specimens by summing the 15-min intervals for which any amount of rainfall was recorded.

When compared on the basis of percent leached per hour of water contact, losses from the immersed small blocks were highest initially but then declined below that of the other leaching methods (Fig 2). Losses of both boron and copper from the LumOut specimens were initially lower than for other methods but became consistently higher during the latter collections. Again, this may reflect the extended periods without rainfall but during which the specimens retained sufficient moisture to allow soluble copper and boron to diffuse from the interior of the specimen to the surface. Similarly, although the LumSim specimens had the fewest total hours of water contact,

their cumulative percent leaching exceeded that of the immersed lumber specimens (Fig 2), presumably because diffusion of soluble components to the wood surface continued between rainfall events.

A comparison of the BlockStir vs BlockNoStir and LumStir vs LumNoStir results indicates that stirring did not cause substantive increases in leaching of boron or copper from either the small cubes or lumber specimens (Figs 1 and 2). The greatest difference observed was actually greater copper release from the BlockNoStir specimens. In previous research, water movement around

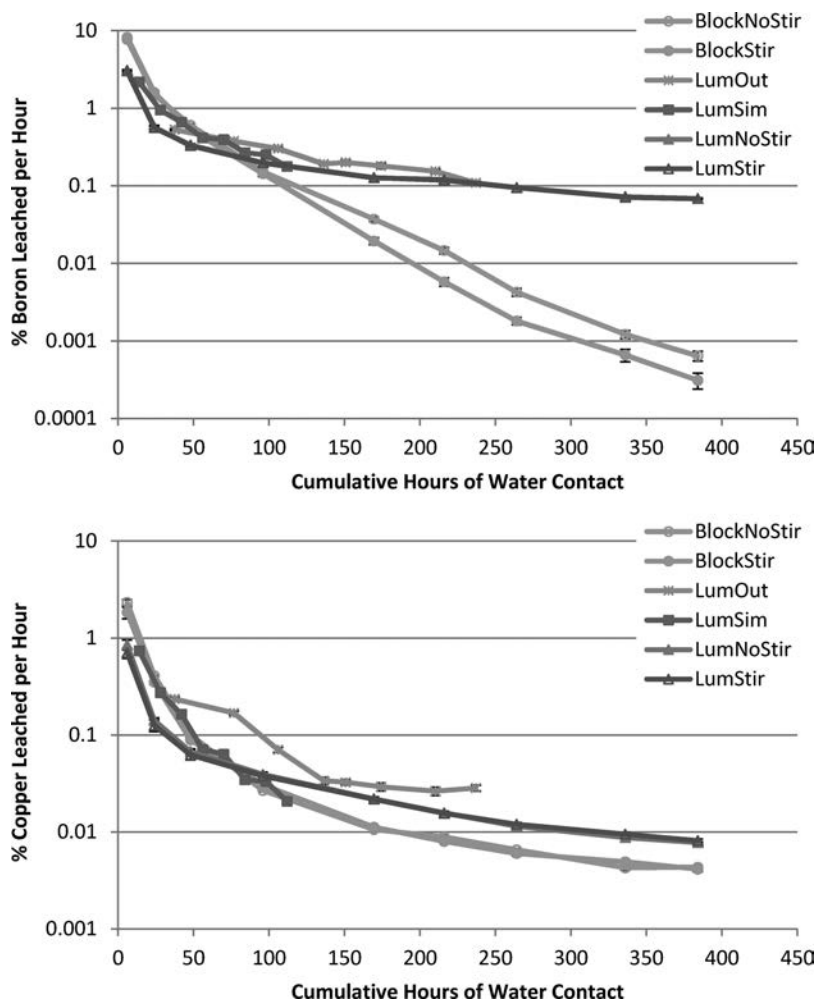


Figure 2. Average percent of boron (upper) or copper (lower) leached from specimens per hour of water contact. Error bars represent one standard deviation from the mean.

wood has been reported to influence leaching, although this effect has not been well quantified. Xiao et al (2002) reported that release of creosote was greatest at the highest flow rate tested and that turbulent flow may have greatly increased leaching. Brooks (2011) suggests that more rapid water movement may increase leaching by promoting water exchange in checks and cracks. Both Xiao et al (2002) and Brooks (2011) used more complex flow-through methods in their evaluations. Van Eetvelde et al (1995) also reported that leaching of copper was greater when using

stirred leaching water than with static leaching trials, whereas also noting that leaching of chromium was unaffected. It is possible that much shorter duration (5 da) of the method used by Van Eetvelde et al (1995) emphasized the effect of stirring in comparison with the more extended AWP method. The AWP standard leaching test specifies the use of a slow stirring speed (eg a tip speed of 250-500 mm/s) (AWPA 2015), but this requirement does not appear to have been based on any specific research. For some laboratories, the stirring requirement may limit the number of

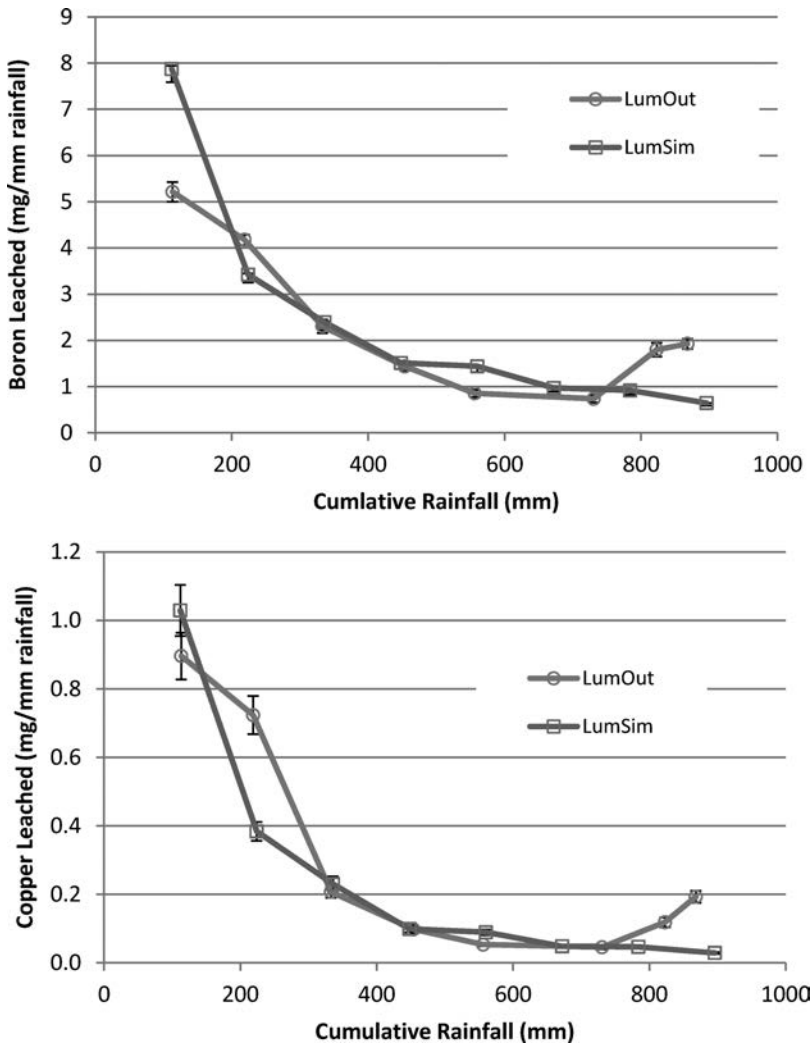


Figure 3. Average milligrams of boron (upper) or copper (lower) leached from specimens per millimeter of rainfall. Error bars represent one standard deviation from the mean.

replicates and/or treatment groups that can be simultaneously evaluated, and the need for this requirement may warrant further consideration.

When compared on a per unit rainfall basis, leaching from the LumSim and LumOut specimens was remarkably similar despite the differences in overall exposure time (Fig 3). Initial leaching, which is strongly influenced by depletion from the exterior of the specimens, did appear to be greater for the LumSim specimens. However, the reverse occurred during the second leaching interval. A notable departure from

the downward trend is observed for the last two collections from the LumOut specimens, when leaching (on a per unit rainfall basis) appears to increase. This increased leaching may be a function of the sustained high MC of the specimens, which remained above 40% during the period preceding the last two leachate collections (Fig 4). The last two exposure periods occurred in the fall, when drying of specimens after rain events may have been slowed because of lower temperatures and less direct sun exposure. The sustained MC above the FSP may have facilitated migration of solubilized boron and copper to the wood

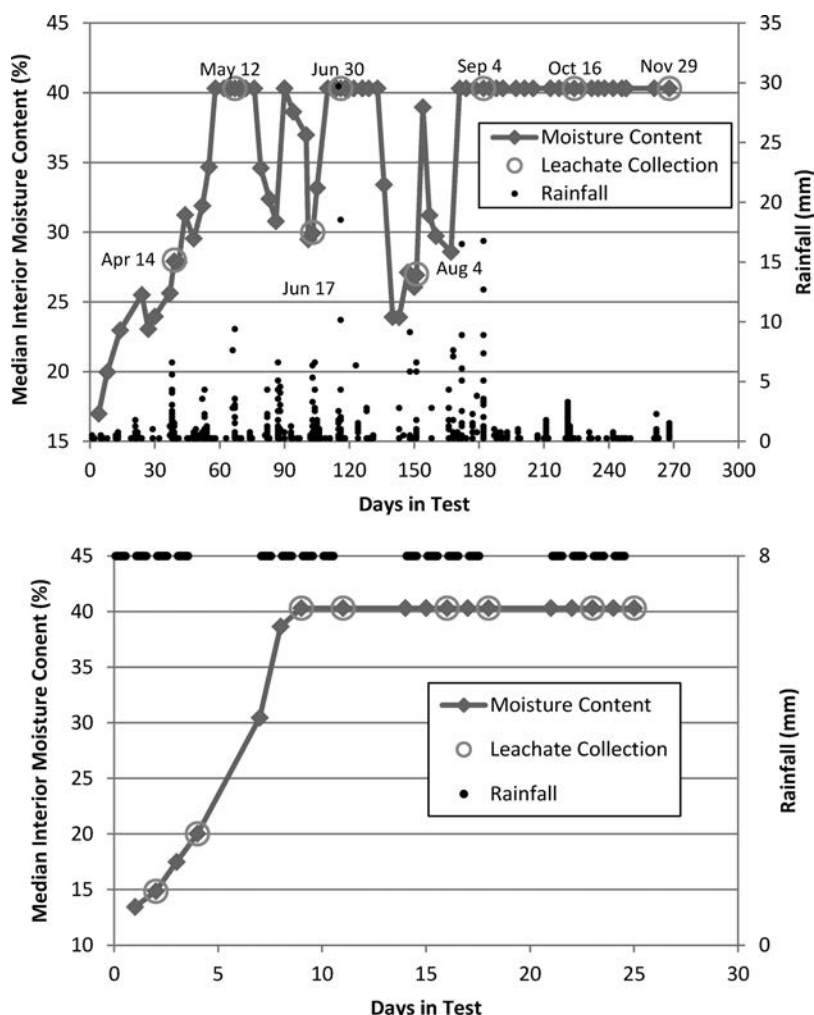


Figure 4. Median interior MC of LumOut specimens during leaching outdoors (top) or LumSim specimens exposed to simulated rainfall (bottom). MC is capped at 40.3% for both sets of specimens because of method limitations.

surface. The rate of rainfall during rainfall events also tended to be greater in the summer than the fall (Fig 4). Previous studies of treated wood exposed to simulated or natural weathering have indicated that both the pattern and rate of rainfall influence the quantity of preservative released. On a per unit rainfall basis, leaching tends to be greater at slower rainfall rates (Cockroft and Laidlaw 1978; Evans 1987; Cooper 2003; Lebow et al 2004), presumably because the wood is wetted for a longer period and a greater proportion of the rainfall is absorbed by the wood.

Of the methods evaluated, the simulated rainfall most closely mimicked the quantity and pattern of copper leaching observed in natural exposure (Fig 3). Simulated rainfall of lumber size specimens appears to have the potential for providing estimates of in-service leaching that are much more useful than those obtained with the current AWP E11 small block method. However, the simulated rainfall method is also considerably more complex than the AWP method, and standardization may not be practical. A method involving immersion of lumber-size specimens would be more practical, and in this study, immersion of lumber specimens under conditions similar to the AWP method did produce boron losses similar to outdoor exposure. Losses of copper, however, were substantially underestimated. It is possible that the relatively short duration of the AWP method did not allow sufficient time for copper to solubilize and diffuse to the wood surface. Rainfall pH and chemical composition may also affect the extent of leaching, depending on preservative characteristics. It is clear that additional research is needed to develop an immersion method that more closely simulates leaching observed in natural exposures.

As a whole, the results of this study reinforce the concept that leaching from wood exposed aboveground is a function of the time that specimens have sufficient moisture to allow diffusion to occur. Although free water in wood is conventionally thought to exist at MSs above the FSP, previous research has found that boron diffusion can occur in wood with MSs as low as

18-20% (Lebow et al 2013). It is likely that concept of FSP as an abrupt transition for diffusion is overly simplistic. As noted by Siau (1995), both free and bound water may be present over a range of MSs below the FSP. Unfortunately, our ability to use this information to predict leaching is limited by the lack of information on the expected range of MSs for wood products exposed aboveground.

CONCLUSIONS

The quantity and pattern of preservative leaching was strongly influenced by leaching method. On a percentage basis, leaching from the small blocks used in the AWP standard method was greater than that of any other method. In contrast, using the same method with larger lumber specimens appeared to underestimate copper losses when compared with leaching observed in an outdoor exposure. It was also noted that stirring, which is currently required in the AWP method, did not appear to increase leaching from either the small blocks or lumber specimens. Of the methods evaluated, lumber specimens exposed to simulated rainfall produced a pattern and quantity of leaching most similar to natural exposure, especially for copper. However, simulated rainfall methods are relatively complex and not well suited for standardization, and additional research is needed to develop simpler immersion methods that more closely approximate leaching in aboveground exposures. The results of this study also indicate that leaching from wood exposed aboveground is a function of the time that specimens have sufficient moisture to allow diffusion to occur. Additional data on typical MSs of wood products in service would be beneficial in development of more realistic leaching methods.

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